



Use of inverse method for bonding quality assessment between bitumen and aggregates under asphalt mixes manufacturing conditions

Sâannibè Ciryle Some, Vincent Gaudefroy, Didier Delaunay

► To cite this version:

Sâannibè Ciryle Some, Vincent Gaudefroy, Didier Delaunay. Use of inverse method for bonding quality assessment between bitumen and aggregates under asphalt mixes manufacturing conditions. Proceedings of the 11th Biennial Conference on Engineering Systems Design and Analysis, TRK 8, Jul 2012, Nantes, France. 9p. hal-00850625

HAL Id: hal-00850625

<https://hal.science/hal-00850625>

Submitted on 7 Aug 2013

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

ESDA 2012 -82361

**USE OF INVERSE METHOD FOR BONDING QUALITY ASSESSMENT BETWEEN
BITUMEN AND AGGREGATES UNDER ASPHALT MIXES MANUFACTURING
CONDITIONS**

Saannibè-Ciryle Somé

LUNAM Institut Français des Sciences et
Technologies des Transports, de l'Aménagement
et des Réseaux (IFSTTAR), Route de Bouaye
CS4, 44344 Bouguenais Cedex, France

Vincent Gaudefroy

LUNAM Institut Français des Sciences et
Technologies des Transports, de l'Aménagement
et des Réseaux (IFSTTAR), Route de Bouaye
CS4, 44344 Bouguenais Cedex, France

Didier Delaunay

Laboratoire de Thermocinétique
de l'École Polytechnique de
l'Université de Nantes, UMR
CNRS 6607 Rue Christian Pauc,
BP 50609 44306 Nantes cedex,
France

ABSTRACT

In roads building, classical asphalt mix manufacturing commonly requires the heating (at 160°C) and the complete drying of aggregates. The induced energy cost has opened the way to develop alternatives processes and materials with low energy/carbon materials such as Warm Mix Asphalt (WMA). In warm mixes processes, aggregates manufacturing temperatures are different and lower than the Hot Mix ones. However, manufacturing temperature reduction can locally lead to poor bonding between bitumen and aggregate during the mixing step, due to the bitumen viscosity increasing, although bonding quality measurement remained a challenge. The aim of our study was to presents two thermal inverse methods for bonding quality assessment. These methods are based on Thermal Contact Resistance (TCR) assessment between bitumen and aggregate, during asphalt mix manufacturing. The experimental test principle consisted of heating both bitumen and cylindrical aggregate to their manufacturing temperatures (over 100°C) and to put them into contact thanks to a special experimental device. According to initial samples temperatures, heat transfer occurs from the bitumen to the aggregate. Two variants of the sequential Beck's method were used to solve the inverse heat conduction problem. The first one consisted of determining the TCR from heat flux and temperatures and the second one

consisted of identifying directly the TCR. The TCR values were interpreted as bonding quality criteria.

Results showed low sensitivity to temperature measurement noise in the second variant of the inverse method. Moreover our study showed that bonding quality depends on bitumen and aggregate temperatures. The higher the component's temperatures, the lower the TCR values and better is the bonding quality.

INTRODUCTION

In roads building, classical asphalt mix manufacturing commonly requires the heating (at 160°C) and the complete drying of aggregates. This operation induces high energy consumption. In the last decade roads companies have developed new processes based on fuel consumption reduction. In these processes coarses aggregates temperatures can be reduced significantly [1-3]. The component's temperatures reduction induces bitumen viscosity increases. However, the effect of this high viscosity on the bonding property between bitumen and aggregates are not well known. In the manufacturing step, hot liquid bitumen comes into contact with hot or warm aggregates. This leads to bonding between the bitumen and aggregates. The influence of bitumen and aggregates temperatures on the bonding quality has not been studied yet at manufacturing conditions. However, some studies

have been carried out at room temperature on cool material manufacture at high temperatures.

Conestrari et al [4] have studied the adhesion/cohesion properties of bitumen-aggregate system and the effect of moisture damage by using mechanical pull-off tests. Varying the temperature of the aggregate surface between 90 and 135°C was expected to influence the adhesion between bitumen and aggregate, and moisture susceptibility. A higher surface temperature should result in higher adhesion values since the lower bitumen viscosity allows better bonding, which should lead to lower sensitivity to water damage. This hypothesis was tested with two types of aggregate (porphyry and limestone). These tests were conducted at room temperature (25°C and 40°C) and some specimens conditioned with immersion. Their studies showed that surface temperature effects were negligible for both aggregate types at 40°C and not negligible in other cases. The difficulties of these tests are the fact that the results are very scattered and need statistical ANOVA treatment to be well understood.

Vasconcelos et al [5] have characterized the adhesion between bitumen and aggregate at different aggregate temperatures by measuring the total energy of adhesion (TEA) using DSC apparatus. Two vials were used, one empty (reference) and the other with 8g of aggregate (sample) consisting of sand of 1.15mm maximum diameter. The aggregates were treated at four different temperatures (90, 110, 130 and 150°C). Bitumen solutions were prepared using 1.5g of bitumen dissolved in 11mL of HPLC grade toluene because the binder in a toluene solution does not compromise the physico-chemical characteristics of the bitumen. Moreover bitumen molecules in a toluene solution have similar kinetics to those of molecules in liquid bitumen at elevated temperatures. This process avoids bitumen heating. The cells were allowed to reach thermal equilibrium. Equilibrium was confirmed as the point when the heat flow ceased to change over time. After reaching thermal equilibrium, the asphalt binder solution was injected with syringes into the vials. The asphalt binder molecules preferentially adhere to the aggregate surface reducing the total energy of the system and producing heat. The heat flow from the reaction cell was recorded over time and the system was allowed to return to thermal equilibrium. The area under the heat flow curve over time was integrated to obtain the TEA. Their results showed that for the non-porous aggregates used, lower aggregate pretreatment temperatures (within the range of 90°C to 150°C) did not significantly impact TEA.

Le Goff et al [6] have investigated the heat transfer that occurs during the solidification of semi-crystalline polymer on a mold surface. On the microscopic scale, the mold surface is not completely smooth and consists of small asperities production from the surface profile. When liquid polymer approaches the mold surface, contact occurs at the peaks of asperities. Rapid cooling at these peaks induces solidification of the polymer to nucleate from these sites. Indeed, it is a location where heat flux density is maximum. They showed that the diameter of the crystallized area increased. The increase of the air gap volume due to the shrinkage during crystallization

increases the Thermal Contact Resistance (TCR). When crystallization is complete, the thermal contact resistance continues to grow at a smaller rate. Just after the contact, its value is relatively high and it decreases to reach a minimum. This decrease is due to the evacuation of the air layer which is trapped in the surface roughness. It takes some time to be evacuated. This principle is quite similar to what occurs during asphalt mixture manufacturing when hot bitumen comes into contact with hot aggregates.

In our study the experimental device used in reference [6] was modified to put hot bitumen into contact with a hot or warm aggregate substrate. The aim of this study is to assess the TCR between both components. As in the case of many studies, [6-13] the interfacial thermal properties such as TCR, surface temperature or heat flux, assessment required inverse heat conduction problem solving. This paper deals with thermal contact resistance between bitumen and aggregate substrates in hot and warm asphalt mixture manufacturing conditions. This TCR was analyzed as a bonding quality indicator.

The sequential method widely described by Beck [16, 17] was used. Two variants of the sequential inverse method were performed to solve the inverse heat conduction problem. The first one consists of determining TCR from heat flux and component's surface temperatures and the second variant consists of direct identification of the TCR.

All methods showed good agreement. However, the first method was more sensitive to the temperature data recorded noisy. Moreover, our study has shown that bonding quality depends on bitumen and aggregate temperatures. The higher the component's temperatures, the lower the TCR values and better were the bond quality.

NOMENCLATURE

C_p	specific heat capacity, $\text{Jkg}^{-1}\text{K}^{-1}$
E	thermal effusivity
Δt	time interval, s
t	time, s
r	number of future time steps
n, k	time discretisation index
m	number of sensors
x	abscissa, m
l	bitumen thickness, m
L	distance between TC5 and TC4, m
Δx	space interval, m
T	average estimated temperature, °C
S	sensitivity coefficient
h	inverse of $\text{TCR Wm}^{-2}\text{K}^{-1}$
J	Residual function
Y	measured temperature, Km^2W^{-1}
R_a	arithmetic average of the absolute values of the measured profile height deviation, μm
TC_i	measured by sensor i
TCR	thermal contact resistance Km^2W^{-1}
<i>Greek symbols</i>	
λ	thermal conductivity, $\text{Wm}^{-1}\text{K}^{-1}$
ρ	density, kgm^{-3}

ϕ heat flux Wm^{-2}
Subscripts
 G,s granular substrate
 b bitumen
 i,j space discretisation index

MATERIALS

Bitumen is a natural polymer of low molecular weight, and behaves as a viscoelastic material. Its microstructure is widely studied by DSC [18, 19]. It is rigid and brittle at low temperature, flexible at room temperature, and flows at high temperature. The bitumen used in this study has the following characteristics: pen grade 35/10 mm and softening point ring and ball temperature 53.6 °C. It has 8mm average thickness and 50 mm diameter.

The aggregate substrate (gneiss substrate) consisted of a cylinder cored with 52mm high and 70mm diameter from a block. Its base surface roughness was characterized by an optical profilometer. The arithmetic average of the absolute value of the measured profile height deviation (Ra) was 75 μm . Four micro-thermocouples, constituted by wires of 80 μm diameter, are placed in the aggregate along its vertical axis. For an accurate implementation of the wires and no disturbance of the heat flux, the substrate was cut along an axial symmetric plane, in two parts. On one of these parts and at different depths from the surface, each wire was soldered on its end on the centre line and placed in a thin parallel groove to the aggregate surface to minimize the heat drain. The exact positions of the sensor junctions with respect to the surface, given in Table 1, were measured by an optical profilometer before the two parts were precisely re-assembled. The first thermocouple which was the most important for the measurement sensitivity respected rather well the conditions described in reference [20].

TC1	TC2	TC3	TC4	Ra
+1.48mm	+1.43mm	+1.48mm	+5mm	75 μm

Table 1: Roughness (Ra define in above paragraph) and thermocouples positions with respect to contact interface

The aggregate and bitumen thermal conductivities were measured according to the guarded hot plate method. In the temperature range of interest 30°C to 160°C, we found a constant value of the thermal conductivity of these samples. The results are given in Table 2.

Differential scanning calorimeter (DSC) was used to investigate the thermal behavior of the specimens. Heating, as defined here, was heating of the samples at a moderated rate by the calorimeter, an average of about 10°C/min up from -80°C to 200°C. The region of interest is from 30°C to 160°C. In this temperature range, the heat capacity was approximated by quadratic polynomial. Fig. 1 and Fig. 2 show DSC results for bitumen, doped bitumen and substrate.

The specific volume was measured by the PvT- α apparatus described in reference [21]. The specific volume was assumed to be linear between 30°C and 160°C as found by the experimental result. The thermal properties are summarized in

Table 2. Apart from specific heat capacity, all other properties of bitumen and doped bitumen are similar.

Thermal properties	Aggregate	Pur bitumen
Thermal conductivity ($\text{Wm}^{-1}\text{K}^{-1}$)	2.59	0.199
Specific heat capacity ($\text{Jkg}^{-1}\text{K}^{-1}$)	$Cp_s(T) = -9 \times 10^{-4} T^2 + 1.512T + 630.37$	$Cp_b(T) = 7.1 \times 10^{-3} T^2 + 2.441T + 1834.7$
Specific volume (m^3/kg)	$v_s(T) = 5 \times 10^{-9} T + 4 \times 10^{-4}$	$v_b(T) = 4 \times 10^{-7} T + 9 \times 10^{-4}$

Table 2: Thermal properties of specimens

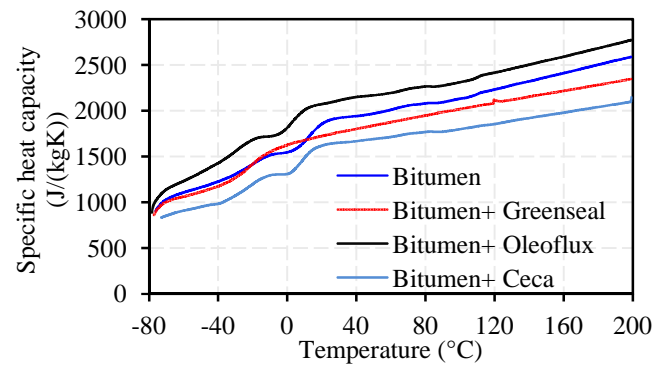


Figure 1: DSC results for bitumen and doped bitumen

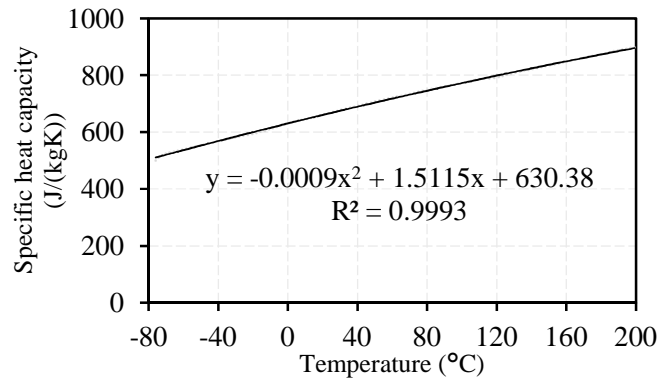


Figure 2: substrate specific heat capacity

EXPERIMENTAL METHODS

The purpose of this study is to investigate the bonding quality when hot bitumen is put into contact with hot or warm aggregate during the mixing operation of asphalt mix manufacturing. In the manufacturing of warm mixes [1-3], bitumen is generally heated to 160°C when aggregates are heated to a temperature range between 100°C and 150°C. This induced a low cooling rate of bitumen during the aggregates coating. The bitumen cooling leads to its viscosity increasing and can limit its penetration in the aggregates microcavities. The consequence of bad aggregate coating is water sensitivity of the pavement at room temperature. Water is known to delaminate the bitumen and aggregates interface. It is then

more important to ensure good bonding between the bitumen and aggregates during mixing operations at high temperature.

However, till now, no experiment has measured the bonding quality during manufacturing step. The experimental study we performed here allowed to study bonding quality in the manufacturing step.

Firts of all, the granular substrate sample described previously was insulated and fixed to plate P0 as shown in Fig. 3 and Fig 4. This plate was equipped with a hot oil circulating system. Then, the top side of the granular substrate is heated by the plate P0. Plate P1 is placed at the substrate bottom. The upper side of this plate P1 contains the same hot oil circulation system as plate P0. This allows heating of the bottom side of the substrate to ensure uniform temperature (T_1) in the substrate sample. The bottom side of plate P1 is equipped with a heater that allows heating of the upper side of the bitumen to temperature (T_2). A plate P2 located at the bottom of the bitumen sample, and equipped with heaters, ensures the bitumen bottom side heating and achieves uniform temperature T_2 . The bitumen sample was placed previously in an elastomer ring. When all temperatures are uniform in each sample, the plate P1 is removed and the granular substrate is suddenly put into contact with the hot bitumen sample. After contact establishment, the heat flux flows through the interface. A better thermal contact leads to an important heat flux flowing through the interface. The bitumen interface temperature decreases whereas the substrate interface temperature increases. Because of the imperfect contact due to the air trapped into the roughness of the substrate and the heat flux constriction, the bitumen interface temperature is different to the substrate interface temperature. We can then define the thermal contact resistance as the temperature gap at the interface over the heat flux at the interface. A small value of TCR means good contact between bitumen and aggregate.

All the experimental tests were carried out using the following conditions: holding pressure: 5 bars, bitumen temperature: always higher than substrate temperature in asphalt manufacturing conditions, contact duration : 60s.

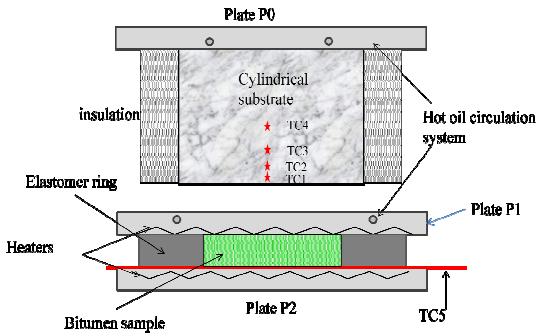


Figure 3: Schematic view of the experimental system before contact establishment

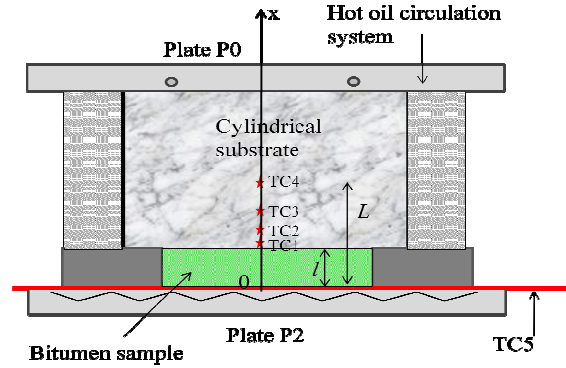


Figure 4: Schematic view of the experimental system after contact establishment

DIRECT PROBLEM FORMULATION

The samples were initially heated to the required temperatures. Afterwards, the aggregate was put suddenly into contact with the hot bitumen. The temperature evolution was measured by the sensors located in the substrate and at the bitumen bottom. The surface temperatures and heat flux assessment lead to a nonlinear inverse heat conduction problem (IHCP) due to the variations of the thermal properties with temperature shown in table 2. For the nonlinear case, the unknowns interfacial temperatures $T_{ss}(l,t)$, $T_b(l,t)$ and the interfacial heat flux $\phi(t)$ need to be determined from the following mathematical formulation of the direct problem in the bitumen and substrate regions between sensors TC5 and TC4.

$$(\rho(T)C_p(T))_s \frac{\partial T(x,t)}{\partial t} = \lambda_s \frac{\partial^2 T(x,t)}{\partial x^2}, \quad 0 < x < l, \quad 0 < t < t_f \quad (1)$$

$$-\lambda_s \left. \frac{\partial T(x,t)}{\partial x} \right|_{x=0} = \phi(t), \quad 0 < t < t_f \quad (2)$$

$$-\lambda_s \left. \frac{\partial T(x,t)}{\partial x} \right|_{x=l} = \phi(t), \quad 0 < t < t_f \quad (3)$$

$$(\rho(T)C_p(T))_b \frac{\partial T(x,t)}{\partial t} = \lambda_b \frac{\partial^2 T(x,t)}{\partial x^2}, \quad l < x < L, \quad 0 < t < t_f \quad (4)$$

$$T(x=0,t) = T_0(t), \quad 0 < t < t_f \quad (5)$$

$$T(x=L,t) = T_L(t), \quad 0 < t < t_f \quad (6)$$

$$T(x,0) = F(x), \quad 0 \leq x \leq L \quad (7)$$

$$TCR(t) = \frac{T_{ss}(l,t) - T_b(l,t)}{\phi(t)} \quad (8)$$

The boundaries conditions $T_0(t)$ and $T_L(t)$ are the temperature history measured by the sensors TC5 and TC4. As additional information, temperatures histories are given by other sensors (TC1, TC2, TC3).

INVERSE ANALYSIS

There is many different ways to solve inverse heat conduction problem (IHCP). Some of them can be found in references [9, 10], [13-17], [22, 23]. In this paper, two inverse methods are used to solve the problem defined by Eqs (1)-(8). Two variants of the sequential Beck's method [9, 13], one using

the interface heat flux estimation and the other one the direct TCR estimation are used to solve the problem.

Surface heat flux assessment

The solution of the boundary problem described by Eqs. (1)-(8) with temperatures and heat flux unknowns can be obtained by minimizing the mean-square deviation discrete functional, described by Eq. (9) under constraints. One assumed the IHCP has been solved up to time t^n ; the estimated heat flux q^n and the temperature field T_i^n are known. Next, the time is advanced one step to t^{n+1} and an estimate q^{n+1} is calculated. Four temperature sensors are located at a different depths x_k below the substrate surface. A temporary assumption is made that heat flux is constant over r future time steps. An estimate is sought of the value φ^{n+1} , constant over r future time steps that minimize the least squares error between the computed and measured temperatures at the sensors locations. The least squares function is expressed as:

$$J(\varphi_{x=l}^{n+1}) = \sum_{k=1}^r \sum_{j=1}^m [Y(x_j, t_{n+k}) - T(x_j, t_{n+k}, t_n, \varphi_{x=l}^n, \varphi_{x=l}^{n+1})]^2 \quad (9)$$

Where $Y(x_j, t_{n+k}) = Y_j^{n+k}$ and $T(x_j, t_{n+k}) = T_j^{n+k}$ are respectively the measured temperature and estimate temperature at the sensors locations. T_j^{n+k} is obtained by solving the direct heat conduction problem Eqs. (1)-(8).

m is the number of sensors located in the substrate (TC1, TC2, TC3).

The value of φ^{n+1} , constant over r future time steps, that minimizes J is sought,

$$\frac{\partial J(\varphi_{x=l}^{n+1})}{\partial \varphi_{x=l}^{n+1}} = 0 = -2 \sum_{k=1}^r \sum_{j=1}^m [Y(x_j, t_{n+k}) - T(x_j, t_{n+k}, t_n, \varphi_{x=l}^n, \varphi_{x=l}^{n+1})] \frac{\partial T_j^{n+k}(\varphi_{x=l}^{n+1})}{\partial \varphi_{x=l}^{n+1}} \quad (10)$$

The last term on the right of Eq. (10) is called sensitivity coefficient. Step function sensitivity coefficient can be written as:

$$S_j^{n+k} = \frac{\partial T_j^{n+k}(\varphi_{x=l}^{n+1})}{\partial \varphi_{x=l}^{n+1}} \quad (11)$$

Expanding the temperature field at the first order in a Taylor series about an assumed heat flux φ^n ,

$$T_j^{n+k}(\varphi_{x=l}^{n+1}) = T_j^{n+k}(\varphi_{x=l}^n) + (\varphi_{x=l}^{n+1} - \varphi_{x=l}^n) S_j^{n+k} \quad (12)$$

Substituting Eq. (12) into Eq. (10), and solving for the desired heat flux,

$$\varphi_{x=l}^{n+1} = \varphi_{x=l}^n + \frac{\sum_{k=1}^r \sum_{j=1}^m [Y(x_j, t_{n+k}) - T(x_j, t_{n+k}, t_n, \varphi_{x=l}^n, \varphi_{x=l}^{n+1}) S_j^{n+k}]}{\sum_{k=1}^r \sum_{j=1}^m (S_j^{n+k})^2} \quad (13)$$

The sensitivity coefficients S_j^{n+k} are the solution of the sensitivity equation formulated as follows:

$$(\rho(T)C_p(T))_b \frac{\partial S(x, t)}{\partial t} = \lambda_b \frac{\partial^2 S(x, t)}{\partial x^2}, \quad 0 < x < l, \quad 0 < t < t_f \quad (14)$$

$$-\lambda_b \frac{\partial S(x, t)}{\partial x} \Big|_{x=l} = 1, \quad 0 < t < t_f \quad (15)$$

$$-\lambda_s \frac{\partial S(x, t)}{\partial x} \Big|_{x=l} = 1, \quad 0 < t < t_f \quad (16)$$

$$(\rho(T)C_p(T))_s \frac{\partial S(x, t)}{\partial t} = \lambda_s \frac{\partial^2 S(x, t)}{\partial x^2}, \quad l < x < L, \quad 0 < t < t_f \quad (17)$$

$$S(x=0, t) = 0, \quad 0 < t < t_f \quad (18)$$

$$S(x=L, t) = 0, \quad 0 < t < t_f \quad (19)$$

$$S(x, 0) = 0, \quad 0 \leq x \leq L \quad (20)$$

We deduce the temperature field $T_j^{n+1} = T(x_j, t_{n+1})$ according to following relation:

$$T_j^{n+1}(\varphi_{x=l}^{n+1}) = T_j^{n+1}(\varphi_{x=l}^n) + (\varphi_{x=l}^{n+1} - \varphi_{x=l}^n) S_j^{n+1} \quad (21)$$

Where T_j^{n+1} are the obtained temperatures from the direct problem solving at abscissa x_j and time step t_{n+1} for q_n heat flux assume.

Eq. (21) allows assessing bitumen and substrate surface temperatures. Using Eq. (13), the TCR can be found according to Eq. (8). For more detailed of this method references [6, 14, 24] can be consulted.

Direct Thermal contact resistance assessment

Eqs. (2) and (3) can be rewritten to introduce the unknown TCR directly. This procedure allows identification of the TCR without surface temperatures and heat flux assessment. The direct problem formulation becomes:

$$(\rho(T)C_p(T))_b \frac{\partial T(x, t)}{\partial t} = \lambda_b \frac{\partial^2 T(x, t)}{\partial x^2}, \quad 0 < x < l, \quad 0 < t < t_f \quad (22)$$

$$-\lambda_b \frac{\partial T(x, t)}{\partial x} \Big|_{x=l} = h(T_m - T_s), \quad 0 < t < t_f \quad (23)$$

$$-\lambda_s \frac{\partial T(x, t)}{\partial x} \Big|_{x=l} = h(T_m - T_s), \quad 0 < t < t_f \quad (24)$$

$$(\rho(T)C_p(T))_s \frac{\partial T(x, t)}{\partial t} = \lambda_s \frac{\partial^2 T(x, t)}{\partial x^2}, \quad l < x < L, \quad 0 < t < t_f \quad (25)$$

$$T(x=0, t) = T_0(t), \quad 0 < t < t_f \quad (26)$$

$$T(x=L, t) = T_L(t), \quad 0 < t < t_f \quad (27)$$

$$T(x, 0) = F(x), \quad 0 \leq x \leq L \quad (28)$$

$$TCR(t) = \frac{1}{h(t)} \quad (29)$$

As described previously, the heat conduction problem is formulated in the least-square sense and consists of determining the optimal solution h that minimizes the function:

$$J(h_{x=l}^{n+1}) = \sum_{k=1}^r \sum_{j=1}^m [Y_j^{n+k} - T_j^{n+k}(h_{x=l}^{n+1})]^2 \quad (30)$$

The minimization procedure remains the same and step function sensitivity coefficient can be written as:

$$S_j^{n+k} = \frac{\partial T_j^{n+k}(h_{x=l}^{n+1})}{\partial h_{x=l}^{n+1}} \quad (31)$$

The corresponding sensitivity coefficients are obtained from the following sensitivity equation:

$$(\rho(T)C_p(T))_b \frac{\partial S(x,t)}{\partial t} = \lambda_b \frac{\partial^2 S(x,t)}{\partial x^2}, \quad 0 < x < l, \quad 0 < t < t_f \quad (32)$$

$$-\lambda_b \frac{\partial S(x,t)}{\partial x} \Big|_{x=a} = T_{in} - T_{ss}, \quad 0 < t < t_f \quad (33)$$

$$-\lambda_s \frac{\partial S(x,t)}{\partial x} \Big|_{x=a} = T_{in} - T_{ss}, \quad 0 < t < t_f \quad (34)$$

$$(\rho(T)C_p(T))_s \frac{\partial S(x,t)}{\partial t} = \lambda_s \frac{\partial^2 S(x,t)}{\partial x^2}, \quad l < x < L, \quad 0 < t < t_f \quad (35)$$

$$S(x=0, t) = 0, \quad 0 < t < t_f \quad (36)$$

$$S(x=L, t) = 0, \quad 0 < t < t_f \quad (37)$$

$$S(x, 0) = 0, \quad 0 \leq x \leq L \quad (38)$$

Similarly, we can deduce $h_{x=l}^{n+1}$:

$$h_{x=l}^{n+1} = h_{x=l}^n + \frac{\sum_{k=1}^m \sum_{j=1}^m [Y(x_j, t_{n+k}) - T(x_j, t_{n+k}, h_{x=l}^n) S_j^{n+k}]}{\sum_{k=1}^m \sum_{j=1}^m (S_j^{n+k})^2} \quad (39)$$

The TCR can be deduced according to Eq. (29).

RESULTS ANALYSIS AND DISCUSSION

Repeatability of the experiment

We have conducted two tests for bitumen temperature of 80°C and substrate temperature of 30°C. The aim was to test the repeatability of measurements. Fig. 5 presents the obtained TCR results. We can note good agreement between the first and the second test for a long time. After 40 seconds small differences can be noted due probably to two dimensions effects that can occur on the insulated surfaces sides. For simplicity of figures the following notation was chosen: B_iG_j. This mean that the bitumen (B) was heated to temperature *i* and the granular substrate (G) was heat to temperature *j*. The discussion of the results is done in the next sections.

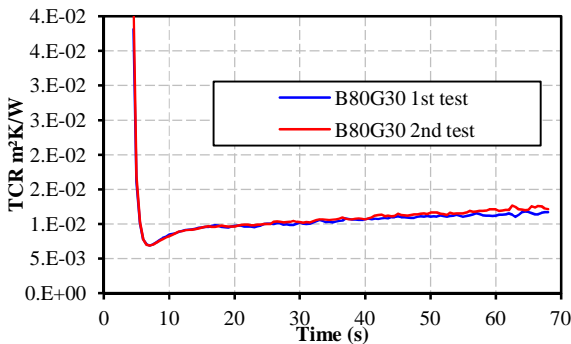


Figure 5: repeatability of measures conducts for B₈₀G₃₀

Methods comparison

The first part of result analysis concerns the comparison of the methods described in the previous sections. For this comparison the substrate and the bitumen was heated respectively to 80°C and 30°C. The surface temperature and heat flux and the TCR were then computed. Figure 6 and figure 7 show respectively the thermal contact resistance and the surface heat flux evolution after the contact. All methods show good agreement between TCR and heat flux results. However, in the first seconds after contact figure 6 and figure 7 shows that Beck's heat flux identification method TCR results are noisy compared to Beck's direct TCR identification method results. This is due to the fact that the TCR assessment from surface heat flux and surfaces temperatures induced an amplification of the TCR noises because it combines the temperatures and heat flux noises.

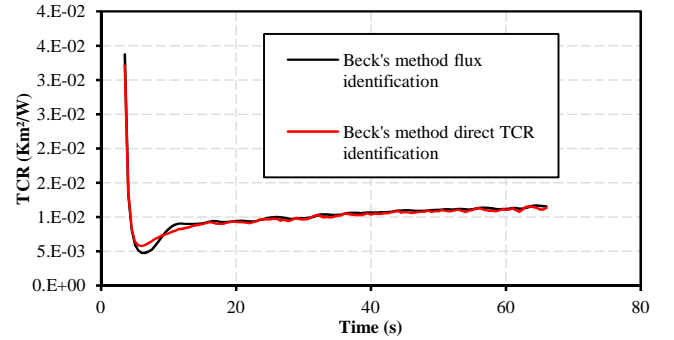


Figure 6: TCR results assessed with heat flux identification and direct TCR identification methods

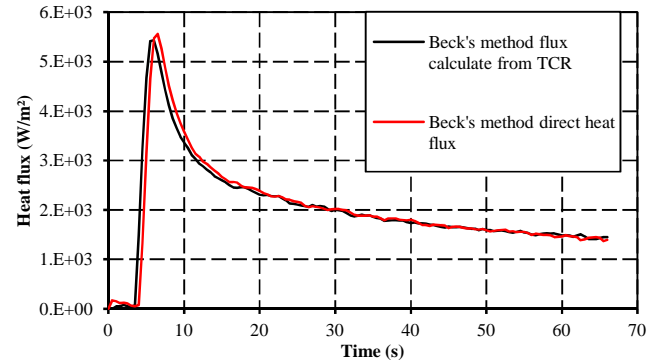


Figure 7: Heat flux results assessed with heat flux identification and direct TCR identification methods

The corresponding surface temperature was computed and represented in figure 8. Weak differences are observed at the first time step as mentioned previously. Figure 8 shows the temperature versus time evolution at the surface of the bitumen and substrate and at the thermocouples location for the B₈₀G₃₀ case.

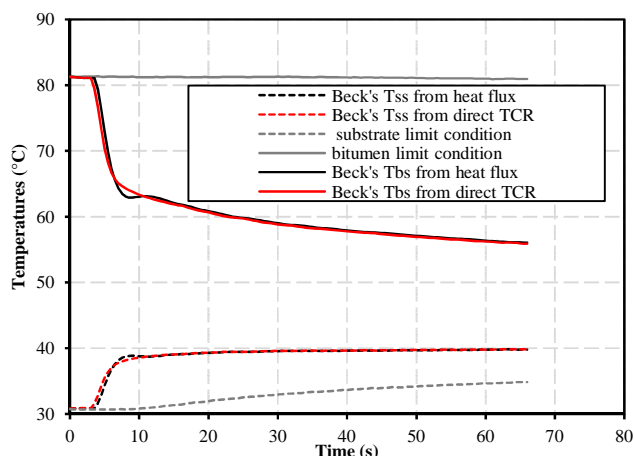


Figure 8: Temperatures profiles results assessed with heat flux identification and direct TCR identification methods

Results analysis

From Fig. 6 result, the following analysis can be done:

After the contact establishment quickly decrease of the TCR is observed. The air is expelled from bitumen-substrate interface causing a high TCR decreasing until a minimal value. Because of roughness of the substrate surface, there is an air layer at the interface between bitumen and substrate. This air gap acts as a resistance. During the first time, TCR depends of substrate roughness, surface tension of the liquid bitumen, wettability and contact angle of the surface, nature of the trapped air and pressure of liquid bitumen. At the end of this stage, the wetted surface and heat flux are maximum. Then TCR is minimum. The TCR is established just on the roughness peaks. Bitumen starts to cool down at these peaks and its cooling leads to its viscosity increasing.

The second step is characterized by the TCR growing. As the substrate's initial temperature is smaller than the bitumen temperature, the contact induces bitumen surface temperature cooling. The bitumen cooling causes its thermal shrinkage which conducts to a slight increasing of the air entrapped in the microcavities at the interface and then increases the TCR value. The thickness of the viscous part of bitumen increases progressively and contracts from the substrate which increases at the same time the size of the interfacial air gap. We assumed that the TCR is totally established when the bitumen surface temperature becomes stationary. The profile of the bitumen surface temperature will be discussed in next section.

Effect of components temperatures

The study of the bonding quality was carried out for several heating cases. The substrate temperature varies from 30°C to 150°C and the bitumen temperature from 80°C to 160°C. Fig. 9 shows the TCR results computed by the Beck's direct TCR identification method. According to Fig. 9, one can note that the increase of the components temperatures induces a decrease of the TCR value. The higher values of TCR are obtained for B₈₀G₃₀. In this case the bitumen is more viscous

and cannot penetrate into the micro-roughness of the aggregate. The smaller values of the TCR are obtained for B₁₆₀G₁₅₀. In this case bitumen is very liquid with low viscosity and this ensures good wettability of the substrate rough surface. In the cases which the substrate temperature is greater or equal to 110°C, the first time TCR values are not very different. However after a long time these values become different due probably to the difference of the thickness of entrapped air. In these experimental tests, the temperatures of some warm mixes are reproduced. We note that, the reduction of the temperature in the warm mixes can induce a loss of bonding quality.

As explain previously, the TCR is assumed to be established when the bitumen surface temperature is constant. These temperatures are represented on Fig. 10. Except B₈₀G₃₀ the others surface temperatures are stationary from 20 seconds and TCR are established.

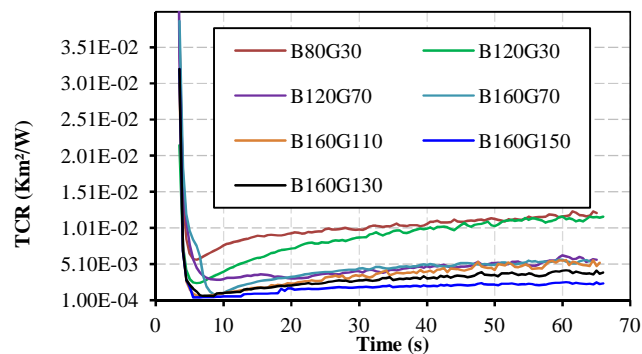


Figure 9: TCR result assess by the Beck's direct TCR method for all bitumen and substrate temperature studied

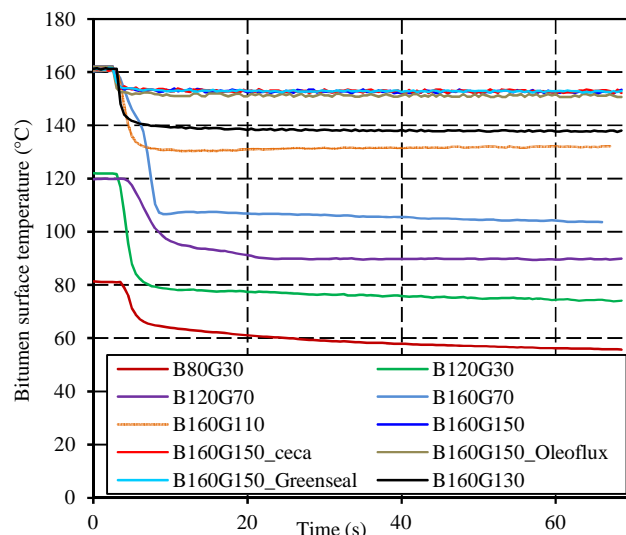


Figure 10: Bitumen surface temperature evolution

Effect of additives

In warm asphalt mix manufacturing, road companies generally add some additives into the hot bitumen before

putting it into contact with aggregates. The additives role is to reduce bitumen viscosity and / or to improve bitumen spreading over the aggregates. Our study proposes to study the effects of some common additives (Ceca, Oleoflux and Greenseal) by TCR measurement. These additives are mixed with bitumen before putting the bitumen in the elastomer ring. They are totally soluble in the bitumen. The effects of the additives in terms TCR are shown in Fig. 11.

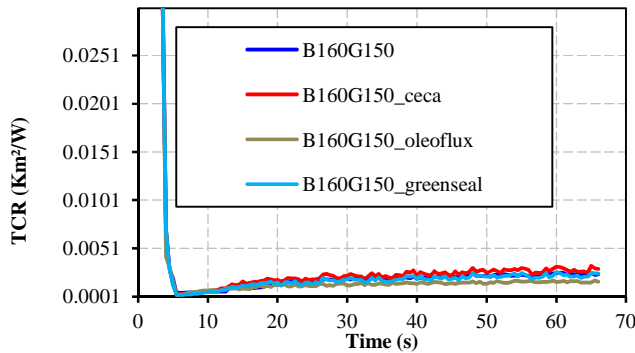


Figure 11: effect of additives on TCR value

The effect of additives was studied for a substrate temperature 150°C and bitumen temperature 160°C. For additives Ceca and Greenseal no significant reduction of TCR can be noted compared to the case B₁₆₀G₁₅₀ without additive. Indeed, studies conducted by Gonzalez et al [1] showed that the Ceca additive dosed at 0.5% did not change the bitumen viscosity while Greenseal additive modifies the bitumen viscosity even at 1% but his effect was not found in our study. In contrast, the Oleoflux additive reduces significantly the TCR value $1.24 \cdot 10^{-3} \text{ Km}^2/\text{W}$ at 20s. This additive is known to modify highly the viscosity, adhesion properties and the mechanical properties of bitumen [3]. The presence of Oleoflux additive in the bitumen, improves the heat transfer rate by minimizing the effect of numerous microcavities filled by air thanks to its surfactant properties. We should mention that the TCR values depend on the roughness of the substrate surface and does not take into account the dynamics due to mixing operation. However, this experimental test has the benefit to assess the bonding quality at the manufacturing period. At 20s the TCR varies from $1.24 \cdot 10^{-3} \text{ Km}^2/\text{W}$ (B₁₆₀G_{150_oleoflux}) to $9.37 \cdot 10^{-3} \text{ Km}^2/\text{W}$ (B₈₀G₃₀). Although these resistances are important, they are consistent with our substrate surface roughness ($R_a=75\mu\text{m}$). We are now working on determining the thickness of the air layer trapped in the substrate roughness. The corresponding bitumen surface temperatures which characterized the TCR establishment are given by Fig. 10.

CONCLUSION

The objective of this paper was to present a bonding quality between bitumen and aggregates assessment method during the asphalt mix manufacturing operation. We proposed to assess the TCR between bitumen and aggregate and then to interpreted TCR as bitumen and aggregate bonding quality indicator in the asphalt mix manufacturing conditions. The results show that the increase of one of the components

temperature (bitumen or granular substrate) induces a decrease of the thermal contact resistance and good bonding between bitumen and aggregate in the asphalt manufacturing operation. The additives generally used by roads companies when components temperatures are reduced to ensure good wettability or low bitumen viscosity effect was studied. We found bonding improvement by the Oleoflux additive, the effect of others is not significant.

One of the main objectives was to make a comparison of the TCR results according to the chosen inverse method. Two methods are used and all of them show good agreement on TCR results. Only the induced noise is different. We suggest finally using Beck's direct TCR identification method to assess the TCR.

ACKNOWLEDGMENTS

We express our gratitude to Nicolas Lefevre and Sébastien Buisson for their crucial participation in the adaptation of the apparatus.

REFERENCES

- [1] J.A. Gonzalez Leon, S. Ligier, P. Caujolle, G. Barreto, Warm mix asphalts with chemical additives: properties and advantages, ENVIRAOD (2009).
- [2] M. Mazé, F. Delfosse, S. F. Dumont, E. Crews, C. Giorgi, Développement en Europe du procédé Tempéra/ Evotherm DAT, RGRA 874 (2009) 50-55.
- [3] E. Beduneau, V. Gaudefroy, F. Olard, C. de la Roche, Enrobés chauds et semi-tièdes EBT : évaluation éformantielle d'une grave-bitume. RGRA 880 (2009) 967-75
- [4] F. Canestrari, F. Cardone, A. Graziani, F. A. Santagata, H. U. Bahia, Adhesive and cohesive properties of asphalt-aggregate systems subected to moisture damage, International Journal of Road Materials and Pavement design EATA (2010) 11-32,
- [5] K. L. Vasconcelos, A. Bhasin, D. N. Little, Influence of reduced production temperatures on the adhesive properties of aggregates and laboratory performance of fine aggregate-asphalt mixtures. International Journal of Road Materials and Pavements Design 11 (2010) 47-64.
- [6] R. Le Goff, G. Poutot, D. Delaunay, R. Fulchiron, E. Koscher, Study and modeling of heat transfer during the solidification of semi-crystalline polymers. International Journal of Heat and Mass Transfer 48 (2005), 5417-5430.
- [7] T. Loulou, E. A. Artyukhin, J.P. Bardon, Estimation of thermal contact resistance during the first stages of metal solidification process: I-experiment principle and modelisation. International Journal of Heat and Mass Transfer 42 (1999) 2119-2127.
- [8] T. Loulou, E.A. Artyukhin, J.P. Bardon, Estimation of thermal contact resistance during the first stages of metal solidification process: II- experimental setup and results. International Journal of Heat and Mass Transfer 42 (1999) 2129-2142.

- [9] P. Le Bot, Comportement thermique des semi-cristallins injectés application à la prédiction des retraits, PhD thesis Université de Nantes 1998.
- [10] A.S. Boutonnet, Étude de la résistance thermique de contact à l'interface de solides déformables en frottement : application aux procédés de forgeage, PhD thesis, INSA Lyon 1998.
- [11] H. Massé, E. Arquis, D. Delaunay, S. Quilliet, P.H. Le Bot, Heat transfer with mechanically driven contact resistance at the polymer-mold interface in injection molding of polymers, *International Journal of Heat and Mass Transfer* 47 (2004) 2015-2027.
- [12] A. Bendada, A. Derdouri, M. Lamontagne, Y. Simard, Analysis of thermal contact resistance between polymer and mold in injection molding, *Applied Thermal Engineering* 24 (2004) 2029-2040.
- [13] Y-C. Yang, W-L. Chen, A nonlinear inverse problem in estimating the heat flux of the disc in a disc brake system, *Applied Thermal Engineering* 31 (2011) 2439-2448
- [14] Bransier, M. Raynaud, A new finite-difference method for the nonlinear inverse heat conduction problem. *Numerical Heat Conduction*, B 9 (1986) 27-42.
- [15] F.C. Cavalcanti, Caractérisation de produit de l'état liquide à l'état solide, PhD thesis, INSA Lyon (2006).
- [16] J. Beck, B. Blackwell, *Inverse Heat Conduction: Ill-posed problems*. Wiley Interscience, (1985)
- [17] N. Özisik, *Heat conduction*, Second Edition, John Wiley & Sons, (1993).
- [18] J.F. Masson, G.M. Polomark, Bitumen microstructure by modulated differential scanning calorimetry, *Thermochimica Acta* 374 (2001) 105-114.
- [19] J. P. Planche, P. M. Claudy, J. M. Létoffé, D. Martin, Using thermal analysis methods to better understand asphalt rheology, *thermochimica Acta* 324 (1998) 223-227
- [20] B. Bourouga, V. Goizet, JP. Bardon, Les aspects théoriques régissant l'instrumentation d'un capteur thermique pariétal à faible inertie. *International Journal Thermal Science* 39 (2000), 96-109
- [21] X. A. Aduriz, C. Lupi, N. Boyard, J.-L. Bailleul, D. Leduc, V. Sobotka, N. Lefèvre, X. Chapeleau, C. Boisrobert, D. Delaunay, Quantitative control of RTM6 epoxy resin polymerisation by optical index determination, *Composites science and technology* 67(2007) 3196-3201
- [22] O.M. Alifanov, *Inverse Heat Conduction Problem*, Springer, Berlin, 1994
- [23] Y. Jarny, D. Maillet, Linear inverse heat conduction problem-two basic examples, *Metti 5 Spring School* June 2011, 1-18
- [24] M.A. Martorano, J.D.T. Capocchi, Heat transfer coefficient at the metal-mould interface in the unidirectional solidification of Cu-8% Sn alloys, *International Journal of Heat and Mass Transfer* 43 (2000) 2541-2552.